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STANDARD OPERATING PROCEDURE

Preparation of Air Sampling Tubes, Resin Jars, and Cartridges

KEY WORDS

Air sampling, charcoal tube, XAD Resin, COC, glass cleaning, packing tubes.

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Environmental Monitoring Branch organization and personnel, such as management, senior scientist, quality assurance officer, project leader, etc., are defined and discussed in SOP ADMN002.

1.0 INTRODUCTION

Many air samplers draw air though a glass, Teflon® or stainless steel cylinder containing a sampling medium capable of trapping the chemical of interest. Possible sampling media include various sizes and types of sorbent resins, charcoal, or filters, used alone or in combination. Sampling cylinders for HI-VOL (high-volume) and LO-VOL (low-volume) air samplers can be prepared from component materials available from various venders (Figures 1, 2 and 5). The cylinders used to hold the sampling media may be glass tubes, Teflon® cartridges, or glass jars. All materials that will come into contact with samples are prepared by washing, double rinsing with deionized water, rinsing with a solvent (pesticide grade isopropyl, ethyl alcohol, or acetone) and low heat drying. A variety of pre-packed sorbent tubes designed for use with the personal air samplers are also available for purchase (Figure 3). Prior to initiating an air study, ensure that the tubes, cartridges or resin jars are of the proper type and size for the sampling medium as well as the analytical lab's requirements. Conducting a trapping efficiency study is one way to verify if the trapping medium traps the analyte(s) of interest (SOP EQAI003.00).

1.1 Purpose

This Standard Operating Procedure (SOP) discusses preparing, labeling and packaging tubes, large cylinders (resin jars) or Teflon® cartridges to be used for air sampling, as well as special Chain of Custody (COC) handling. This SOP will describe methods for packing tubes or resin jars with trapping media (such as XAD resin). Study specific decisions may be made by the project leader and chemist regarding sample tube/jar/cartridge preparation. These decisions should be described in the study protocol.

1.2 Definitions

1.2.1 **XAD** resin is a styrene-divinylbenzene polymeric adsorbent resin available from Rohm and Haas Company. Read the XAD MSDS prior to working with the resin.

1.2.2 A **Chain of Custody (COC)** is a legal document designed to track a sample container from container preparation through sample analysis as defined in SOP ADMN006.00.

2.0 MATERIALS

2.1 HI-VOL sample jars:

a) Appropriate resin, usually XAD resin, cleaned by laboratory
b) Glass cylinders (called HI-VOL resin jar)
c) Super fine, stainless-steel screen cut to fit resin jars (100 to 120 mesh, small enough so that no resin will pass through, approximately 3 5/8 in. diameter)
d) Open-cell, foam sponge cut 1 inch larger than resin jar inside diameter
e) Two expanded metal stainless-steel screen cut to fit resin jars (0.050 in. thick, 0.50 in. corrugated, approximately 3 5/8 in. diameter) (Figure 4)
f) Aluminum foil
g) Half-pint Mason jar
h) Gaskets (approximately 3 5/8 in. I.D., 4 3/8 in. O.D., 3/16 in. thick)
i) Low pressure, high capacity glass fiber filter paper (8in. by 10 in) (Pall Corporation, Ann Arbor, MI, product number 61638)
j) Office folder to store filters
k) Glass graduated cylinder (200 to 300 ml)

2.2 LO-VOL sample tubes:

a) Appropriate resin, usually XAD resin, clean by laboratory
b) Glass tubes (called LO-VOL resin tubes, with indentation in glass)
c) Super fine, stainless-steel screen cut to fit tubes (100 to 120 mesh, small enough that no resin will pass through)
d) Glass wool
e) Two Rubber stoppers that fit glass tubes
f) Plastic packaging tape
g) Aluminum foil
h) Forceps
i) 25 ml glass beaker
j) 200 ml glass or plastic beaker
k) Scale that reads to two decimal places
l) Scissors
m) Stainless steel rod or spatula at lease six inches long

2.3 Pre-packed sorbent tubes (packed by the manufacturer):

a) Pre-packed sorbent tubes (Figure 3)
b) Tygon® tube (R-3603) cut into 1-inch length pieces
c) Tube caps (2 per tube)

2.4 Teflon® Resin Cartridges for use with LO-VOL samplers

a) Teflon® cylinder (12.2 cm height, 4.1 cm I.D.)
b) Teflon® top & bottom cap
c) Two 100-mesh stainless steel screen, cut to fit cylinder
d) One 100-mesh screen to fit inside of Teflon® connector cap
e) Two Teflon® screens (0.7 cm height, 4.0 cm O.D.)
f) 30 ml bulk of washed XAD4 resin
g) Washed Teflon® extruder (longer than cylinder, for pressing screens into place)
h) Teflon® pipe tape
i) Aluminum foil
j) 50 ml glass beaker
k) 500ml glass or plastic beaker
l) Scale that reads to two decimal places
m) Zip ties with sample tags

2.5 Labels pre-printed with Study Number, Sample Number (A and B if linked tubes in series), and Sample Type (air).

2.6 Clear adhesive tape wide enough to cover a label.

2.7 COCs appropriate to the study.

2.8 Small (minimum 6 X 12 inches) resealable plastic bags.

2.9 Shipping box or archive box

2.10 Permanent ink pen.

2.11 Latex gloves

3.0 PROCEDURES

3.1 HI-VOL Jar Preparation (Figure 1)

Hi-V jars are used with high volume samplers (Kurz Instruments, Inc.). See SOP EQAI003.00 for instructions for the use of a Kurz Instruments, Inc. high volume sampler.

3.1.1 The resin must be cleaned prior to use with a solvent compatible with the chemical analytical method and dried. The procedure takes about one week and is conducted by the California Department of Food and Agriculture Laboratory Services for the Environmental Monitoring (EM) Branch.

3.1.2 Clean glass HI-VOL resin jars according to SOP EQOT002.00, then rinse with a solvent specified by the lab (either pesticide grade isopropyl or ethyl alcohol, or acetone) and heat-dry in an oven at a low temperature (90°C). Note: do not heat dry glassware rinsed with a solvent at a temperature over 90°C.

3.1.3 Gather materials listed in 2.1 above. Wash, double rinse with deionized water, rinse with the same solvent used to wash glass in 3.1.2 and heat-dry the stainless steel screen and the two expanded metal screens in a low temperature (90°C) oven. Rinse the graduated cylinder with the same solvent and dry before using.

3.1.4 While wearing latex gloves, begin assembling the HI-VOL resin jar by placing one of the expanded stainless steel screens on the inside lip near the bottom of the jar. This screen holds all of the following materials in place.

3.1.5 The next three pieces are stacked outside of the jar. Center a piece of fine stainless-steel screen on top of the sponge then place the second piece of expanded screen on top of the fine screen.

3.1.6 Place the whole assembly with the expanded metal screen on top into the top of the resin jar so that it rests on the expanded metal screen already in place. The open cell sponge should cup the fine screen and top expanded screen and hold it in place.

3.1.7 Wrap the outside of the jar with aluminum foil to prevent photolysis of the analyte to be trapped on the resin. Secure with adhesive tape.

3.1.8 Follow instructions in the section 3.5.3 for packaging and labeling of HI-VOL jars.

3.1.9 While working under an operating vacuum hood, measure 125 ml of cleaned XAD resin with a cleaned graduated cylinder and pour into a new half-pint mason jar. Tap the graduated cylinder to

remove as much resin as possible. Replace the mason jar lid. The resin will be poured from the mason jar into the HI-VOL jar after the HI-VOL jar is placed on the sampler at the field site. Be careful not to spill the resin, it is very slippery. (The EM Branch has used 125 mls of XAD resin in all studies using the HI-VOL air pumps. A different volume of resin may be considered. However, as with any change a trapping efficiency study should be conducted (See SOP FSAI003.00).

3.2 LO-VOL Resin Tube Preparation (Figure 2)

The LO-VOL resin tube is used with the Anderson® air sampler or any sampler that operates at 1 to 30 liters per minute.

3.2.1 Clean glass LO-VOL resin tubes according to SOP EQOT002.00, and then rinse with a solvent and heat dry as described in 3.1.2.

3.2.2 Gather materials listed in 2.2 above. Wash, double rinse with deionized water, rinse with a solvent (pesticide grade isopropyl or ethyl alcohol, or acetone) and heat dry (90°C) the stainless steel screens. Rinse the beakers, rod/spatula, scissors and rubber stoppers with the same solvent and air dry before using.

3.2.3 While wearing latex gloves, begin assembling the LO-VOL tube by placing a piece of super fine stainless-steel screen with forceps into the bottom of the tube against the indentations in the glass (Fig. 2). The indentations retain the materials in the tube during sampling. Next place a 1 cm plug of glass wool with forceps above the screen within the LO-VOL tube.

3.2.4 While working under a vacuum hood, tare the weight of a 25ml beaker on a scale, measure 15 mls of resin with a 25 ml beaker, and record the weight of the resin in grams to two decimal places. This will be the amount of resin added to each tube. The weight of the 15 ml of resin must be determined daily because the resin absorbs water from air and may lose solvent by evaporation. (The EM Branch has used 15 mls of XAD resin in all studies using the LO-VOL resin tubes. A different volume of resin may be considered. However, as with any change a trapping efficiency study should be conducted (See SOP FSAI003.00).

3.2.5 Place a 200ml plastic or glass beaker on the scale. Place a LO-VOL tube as assembled in 3.2.3 into the beaker and tare the weight. Pour the resin from the 25 ml beaker into the LO-VOL tube on top of the glass wool until the resin weight reaches the weight determined in 3.2.4.

3.2.6 With forceps or a rod, place a 1 cm plug of glass wool on top of the resin.

3.2.7 Wrap rubber stoppers with plastic tape such as packaging tape material to minimize absorption of pesticides to the stoppers. Insert the wrapped stoppers into both ends of the LO-VOL tube.

3.2.8 Wrap the glass tube, but not the stoppers, with aluminum foil and secure with adhesive tape.

3.2.9 If the LO-VOL tubes will not be used right away, place about 10 tubes in a plastic bag. Label the bag with the date, type of resin used, the date the resin was washed and the solvent used to wash it. Store in a clean dry place at room temperature, and away from volatile chemicals. . Prior to field use, follow instructions in sections 3.5 and 3.6 for packaging and labeling of LO-VOL tubes.

3.3 Pre-packed (manufactured) tubes preparation

Pre-packed tubes can be purchased for a variety of air samplers. EM generally purchases pre-packed tubes for low volume air samplers such as the SKC Air Chek Samplers.

3.3.1 Cut Tygon® tube listed in Section 2.3 above into 1-inch lengths. Follow instructions in the section 3.6.2 for packaging and labeling of pre-packed tubes.

3.3.2 Check protocol to see if and how many breakthrough tubes are necessary.

3.4 Teflon® resin cartridge preparation (Figure 5 and 6)

3.4.1 Some laboratories prefer Teflon® resin cartridges over using glass resin tubes. Teflon® resin cartridges are used with the Anderson® air sampler or any sampler that operates at 1 to 30 liters per minute. The Teflon® resin cartridges contain more resin than the LO-VOL glass tubes and have a larger opening.

3.4.2 Clean the Teflon® cylinder, caps and screens by washing them with soap and water, rinsing with deionized water and a finally rinsing with acetone.

3.4.3 Gather materials listed in 2.4 above.

3.4.4 While wearing latex gloves, begin assembling the Teflon® resin cartridge by placing a 100-mesh stainless steel screen on the concave side of the Teflon® screen.

3.4.5 Locate the top (Figure 5) of the Teflon® cylinder (with the outside grips). Place the Teflon® Screen assembly inside and flush with the top of the Teflon® cylinder with the Teflon® screen positioned on the top.

3.4.6 Press the screen in all the way to the bottom of the recess with the Teflon® extruder to make sure the screen is in place.

3.4.7 While working under a vacuum hood, tare the weight of a 50 ml glass beaker on the scale, then measure out 30 ml bulk of washed XAD4 resin into the beaker, record the weight of the resin in grams to two decimal places. This will be the amount of resin added to each tube. The weight of the particular volume of resin must be determined daily because the resin absorbs water from air and may lose solvent by evaporation. Note: EM has used 30 ml of resin in the cartridges. More or less resin may be used if verified by a trapping efficiency study (SOP FSAI003.00).

3.4.8 Place a 500ml plastic or glass beaker on the scale. Place the Teflon® cartridge as assembled in 3.4.4 through 3.4.6 into the beaker and tare the weight. Pour the weighed resin from the 50 ml beaker into the cartridge on top of the screen until the resin weight reaches the weight determined in 3.4.7.

3.4.9 Tap the Teflon® cylinder gently on the table to even out the resin.

3.4.10 Repeat step 3.4.2 to prepare another Teflon® screen with a 100-mesh screen.

3.4.11 Then, place the 100-mesh screen side of the screen assembly towards the resin that is inside of the Teflon® cylinder. Hold the cylinder on the table and push the screen assembly #2 into the cylinder with the Teflon® extruder until it rests levelly on the resin bed.

3.4.12 After making sure the screens are secured, wrap threads on resin side to cylinder with Teflon® pipe tape and cap with a Teflon® connector cap.

3.4.13 Invert the cylinder, wrap a piece of Teflon® tape around the threads and cap the cylinder with the other Teflon® cap.

3.4.14 Finally, cover the opening of the connector cap with a small 2 x 2 inch foil piece. Prior to field use, follow instructions in sections 3.5 and 3.6 for packaging and labeling of Teflon® resin cartridges.

3.5 Labeling Tubes, Jars and Cartridges

3.5.1 Obtain sufficient sample jars/tubes/cartridges, caps/stoppers/lids, labels, and COCs to complete the number of sample jars/tubes/cartridges required by the study. Labels need to be printed with the study number, unique sample number (see 3.5.2 for tubes in series), and sample type (air) for every tube that will be used. A spreadsheet program works well for making labels.

3.5.2 Sampling tubes are often used in series to check for break-through of analyte. For example, in the case of the pre-packed SKC® charcoal tubes, each tube is labeled with the same sample number. However, one tube (usually the 400 mg charcoal tube) is marked A and the other (200 mg tube), B. In some cases a third tube or tubes with different trapping medium may be used. Tube A is considered the primary sample and B the break-though sample used to check if the primary tube trapped all of the analyte in the air stream. The number of tubes and type of tube selected should be stated in the study protocol. Also, if photolysis is considered a

problem for the analyte, wrap each jar/tube/cartridge with aluminum foil and secure with tape.

3.5.3 Cut out a pre-printed label and affix it lengthwise to the tube or horizontally on the resin jar, on top of the foil wrap if used, using clear tape. Smooth the adhesive tape to assure a good seal around the label.

3.5.4 Teflon® cartridges are usually labeled by using a zip-tie with a sample number on an attached tag. Adhesive tape will not stick to the Teflon® cartridge.

3.6 COC handling and packaging of jars, tubes, and cartridges

3.6.1 Fill out the COC for each jar, cartridge, sample tube or set of tubes as detailed in SOP ADMIN006.00. Generally, only one COC is needed for tubes in series. At a minimum, each COC must have the study number, sample number, chemicals to be analyzed, and the preparer's signature.

3.6.2 For both tube types, LO-VOL and pre-packed fold the COC in half and place in a 6" x 12" plastic bag. Place the tube or tube set in the plastic bag with the COC. For the pre-packed tubes, also place 2 caps per tube and 1 piece of Teflon® tube (2 Tygon® for 3 tubes) in the plastic bag and seal.

3.6.3 For resin jars, the COCs should be filled-out as in 3.6.1. The resin jars should be placed in a protective box with dividers. The box should be labeled with the date the chemist washed the resin and the type of solvent used to wash the resin. Then the COCs matching the HI-VOL jars should be simply stacked on top of the jars inside the box in sample number order. Also place two gaskets per HI-VOL jar into the box for use with the HI-VOL sampler in the field. One filter paper for each HI-VOL jar should be placed in an office folder to be stored with the HI-VOL jars. The glass fiber filters are placed under the jar during sampling to maintain correct air-flow (SOP EQAI003.00).

3.6.4 For cartridges, the COCs should be filled-out as in 3.6.1. The COCs can be either placed in a box or bag with the cartridges.

3.7 Storage

All prepared resin jars, sample tubes, and cartridges with COCs for a given study should be stored together in archive-boxes or storage-boxes on the shelves in the EM warehouse. The boxes with resin jars and cartridges should be placed in a plastic bag to prevent them from accumulating dust. The boxes should be stored indoors. Label the box with the study number and date prepared to make identification easier. Sealed pre-packed tubes should last indefinitely if stored indoors at room temperature. Resin tubes, jars and cartridges packed by EM should be emptied, washed and repacked after one year. Resin tubes, jars and cartridges packed by EM can be used beyond a year after packing if a partial trapping efficiency study is conducted to ensure that the resin is still trapping the same percentage of analyte during the original study and that there is no contamination of the tubes. Washed resin stock remaining in the solvent bottle can be used to pack tubes for up to two years.

4.0 STUDY SPECIFIC DECISIONS

If deviations from the standard operating procedures outlined above are required, they should be detailed in the study protocol. Before preparing any air sampling protocol for a study, the project leader or field coordinator must determine:

4.1.1 The type of air sampling device that will be used (high or low volume, or personal air sampler).

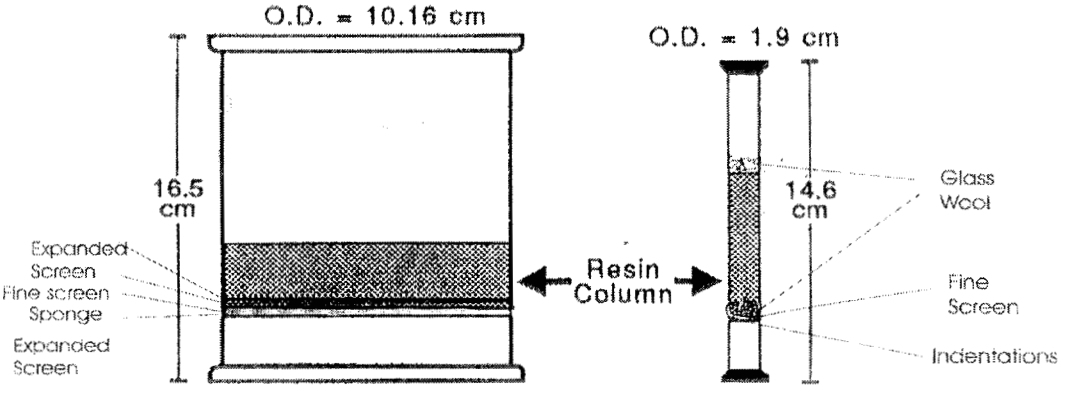
4.1.2 The type of jar, cartridge or tube appropriate for the air sampler.

4.1.3 The type of resin or trapping medium.

4.1.4 If break-through may be a problem, use tubes in series or adjust sampler flow volume.

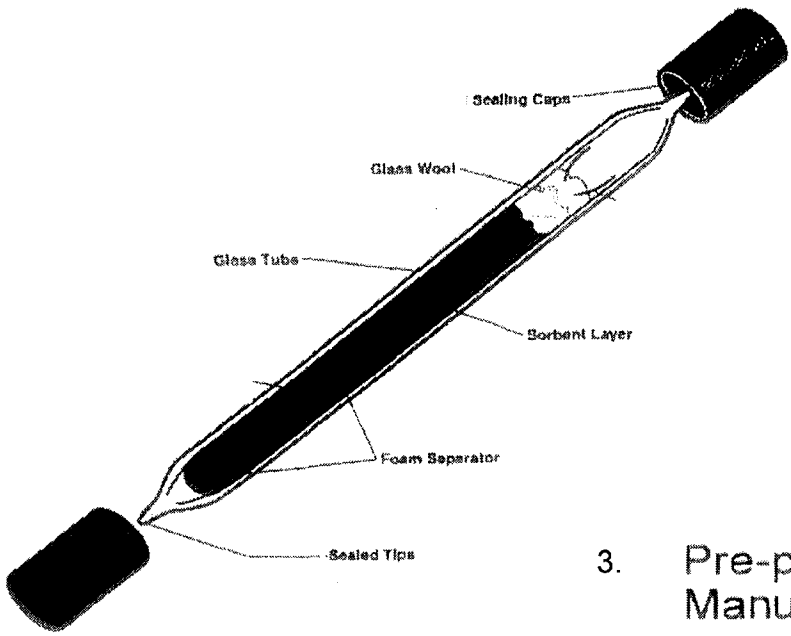
4.1.5 The number of jars, cartridges or tubes required including spares and quality control.

4.1.6 Transport and storage arrangements.



1. HI-VOL
Resin Jar
(125 ml Resin)

2. LO-VOL
Resin Tube
(15 ml Resin)



3. Pre-packed or Manufactured Tube

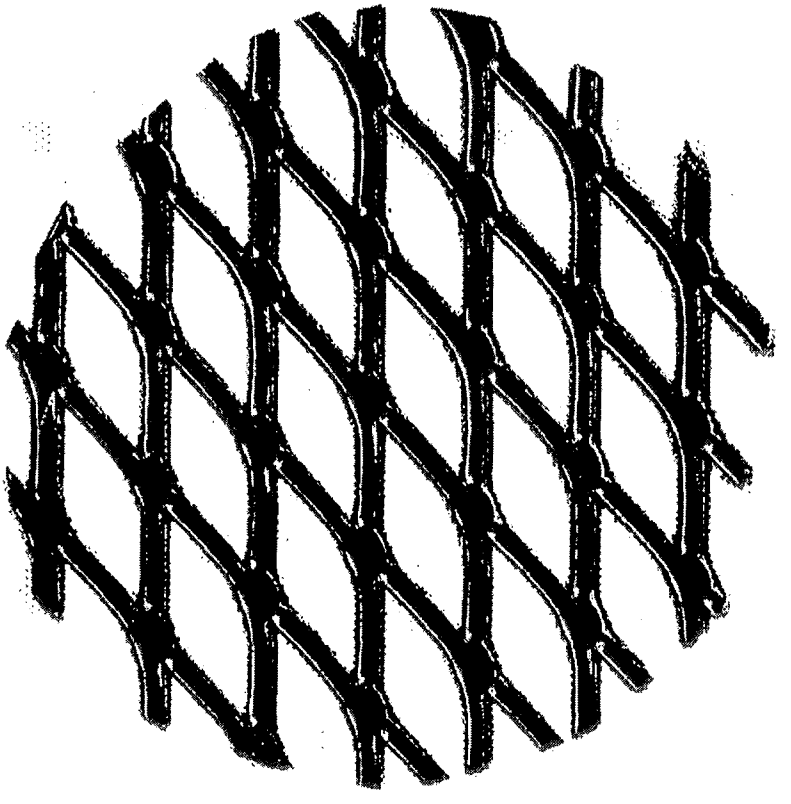


Figure 4. Expanded stainless-steel screen

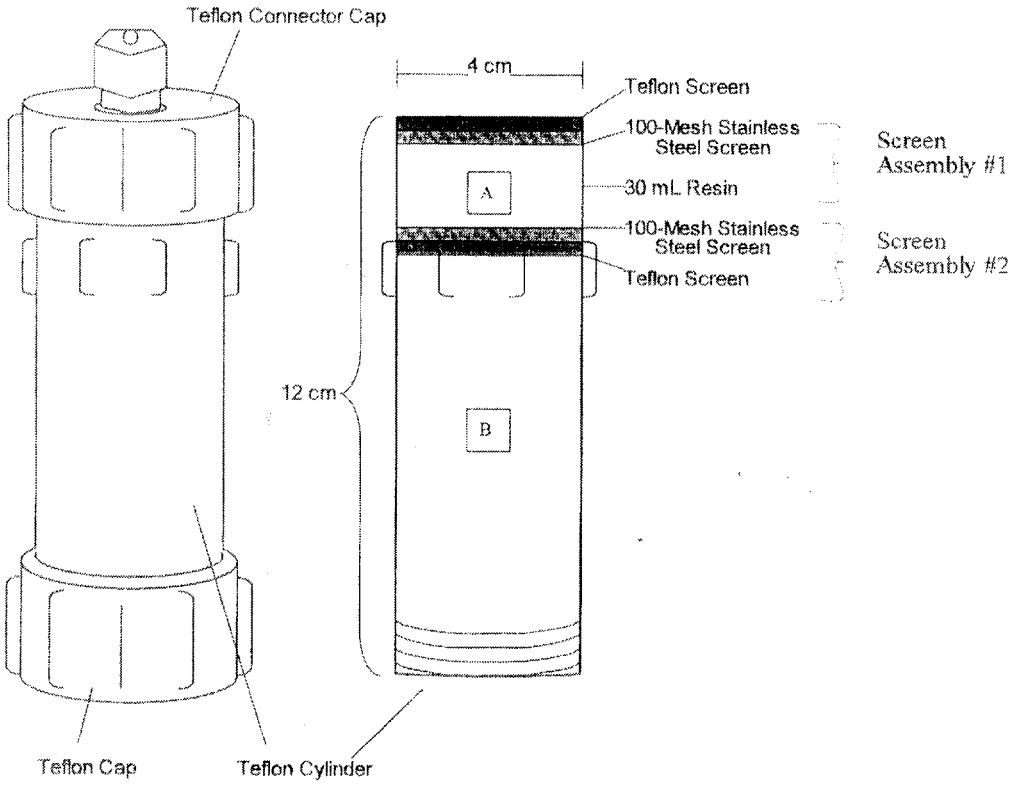


Figure 5. Teflon® Resin Cartridge
(30 mL Resin)

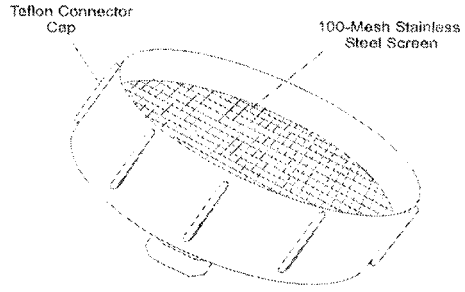


Figure 6. Teflon® Connector
Cap Assembly